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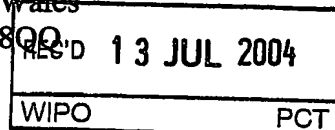


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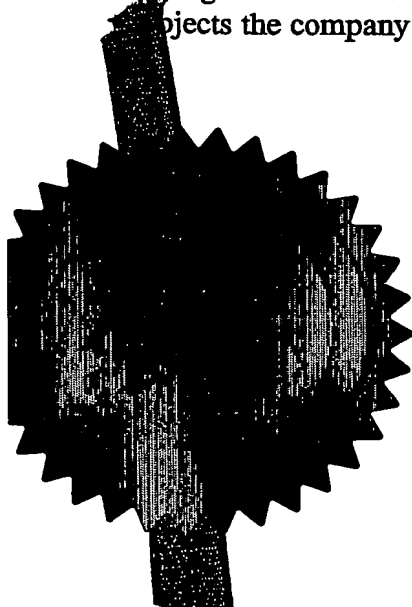
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Signed

*Stephen Hordley*

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07APR03 E798287-1 D01091  
P01/7700 0.00-0307975.3

# Request for grant of a patent

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## 1. Your reference

CI 1616 GB

## 2. Patent application number

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0307975.3

5 APR 2003

## 3. Full name, address and postcode of the or of each applicant (underline all surnames)

Imperial College Innovations Ltd.,  
South Kensington Campus, Imperial College London,  
London SW7 2AZ. 8214173005

Patents ADP number (if you know it)

and

Cambridge University Technical Services Ltd.,  
The Old Schools, Trinity Lane,  
Cambridge CB2 1TT.

If the applicant is a corporate body, give the country/state of its incorporation

6956809007

## 4. Title of the invention

COMPOSITE STRUCTURE

## 5. Name of your agent (if you have one)

"Address for service" in the United Kingdom  
to which all correspondence should be sent  
(including the postcode)

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JOHNSON MATTHEY TECHNOLOGY CENTRE  
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READING RG4 9NH

Patents ADP number (if you know it)

FS1177  
11/04/03

7258312001

125001

## 6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number  
(if you know it)

Date of filing  
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## 7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

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## 8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

a) any applicant named in part 3 is not an inventor, or

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Claim(s)	3
Abstract	1
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Priority documents

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Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

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One 

11.

I/We request the grant of a patent on the basis of this application.

Signature

  
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Date

4 Apr 03

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## COMPOSITE STRUCTURE

This invention relates to composite structures, in particular to composite  
5 structures containing conductive organic species.

Composite structures are known, for example in the formation of electrochemical  
devices such as solar cells. A particular example of a thin-film solar cell application is  
the dye-sensitised cell developed by Grätzel *et al.* (*Nature*, 1991, 353, 737), where a  
10 high-surface area, dye-coated semiconducting working electrode is in contact with a  
charge-carrying, mobile redox couple or hole-transporting material (htm). The action of  
the redox couple or htm is to complete the charge transfer process by injecting an  
electron into the photo-oxidised dye to restore it to the ground-state. In early work, cells  
were made with the redox couple dissolved in a liquid electrolyte. More recently,  
15 increasing efforts have been made to find solid-electrolyte alternatives, for example by  
incorporating gelling agents or organic polymers (Grätzel *et al.* *Nature*, 1998, 395, 583).

To improve the amorphous character, and hence conductivity, transparency etc.,  
of these types of materials, spiro versions of triarylamine have been developed  
20 e.g. spirobifluorene triarylamine derivatives (U. Bach *et al.*, *Adv. Mater.*, 2000, 12, 1060;  
Kruger, *et al.*, *Adv. Mater.*, 2000, 12, 447). Furthermore, triarylamine materials  
incorporating ion-chelating structures have been found to function as hole-transporting  
materials in Grätzel-type cells (WO 02/051958).

25 In Grätzel-type cells, a mobile ionic species needs to be added to the organic htm  
in order to balance the electronic charge generated on illumination of the semiconductor.  
Normally, a lithium salt in a pyridine-based solvent is used as the ionic species (Grätzel  
*et al.* *Nature*, 1998, 395, 583). Such solutions of salts can be hazardous, and being  
mobile, volatile phases, they are problematic to contain within the cell. A typical quasi  
30 solid-state version of the Grätzel cell thus comprises a dye-sensitised titania layer, coated  
with a mixture of a hole-conducting spiro polymer blended with a lithium salt and tert-  
butyl pyridine. The two outer surfaces of the cell usually carry a conducting metallic or  
oxide layer to extract current from the cell. It is possible to omit the mobile ions,  
however, this severely limits the cell efficiency.

The present applicants have found that by confining an ionic species to the interfacial region between a conducting electrode and a conducting polymer, the problems associated with the use of a mobile species can be mitigated.

5 In accordance with a first aspect of the present invention, a composite structure comprises a dual-function material intermediate a conducting material and a semiconductor; wherein the dual-function material comprises an organic material and at least one ionic species; wherein the organic material has both electronic charge transport properties and supports or chelates the at least one ionic species; and wherein the  
10 conducting material comprises an ohmic conductor, a semiconducting material or an ionic conductor.

The present invention provides a significant advantage over for example, the Grätzel type cell, in that the dual-function material effectively confines an ionic species  
15 at its interface with a semiconductor, facilitating charge transfer at this interface. Problems associated with leakage and migration of liquid phase, such as a solution of a lithium salt are avoided.

The ionisation potential and/or the electron affinity of the organic constituent of  
20 the dual-function material should be such that it favours ordering of the ionisation potential and/or electron affinity relative to the semiconductor, enabling charge separation across the interface. The dual-function material may also serve to reduce any interfacial energetic mismatch between the conducting material and the semiconductor.

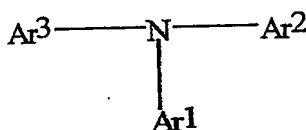
25 Preferably, the organic material comprises at least one moiety represented by the general formula (I):



(I)

30 wherein [Y] comprises an organic semiconductor; and wherein X comprises an ion-chelating group.

The present applicants have discovered a novel class of hole conducting polymers, which can also display electronic conduction properties. These polymers, which are based on tri-aryl amine moieties, are detailed in WO 02/051958 and comprise ion-chelating side-chains which can support or chelate ionic species, thus providing the required ionic component. Thus in a preferred embodiment, [Y] comprises a moiety represented by the general formula (II):



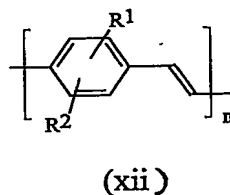
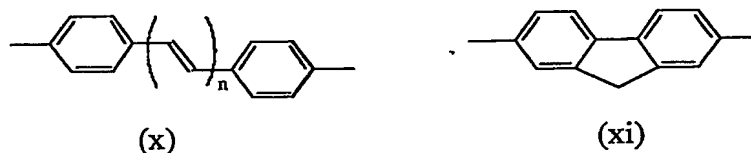
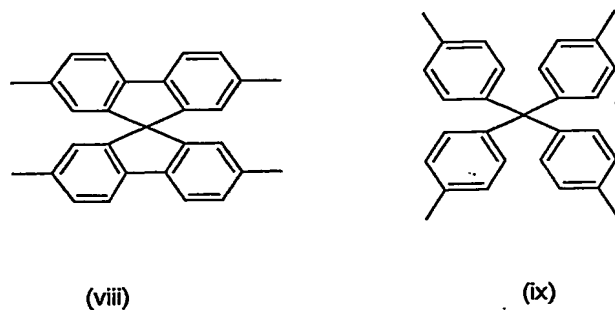
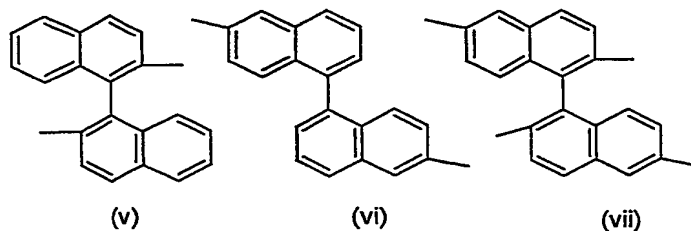
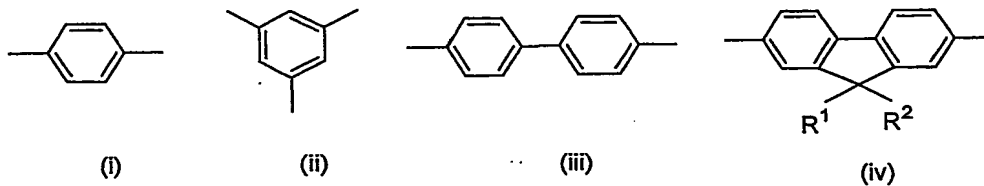
(II)

wherein  $\text{Ar}^1$ ,  $\text{Ar}^2$  and  $\text{Ar}^3$  are independently substituted or unsubstituted aromatic or hetero-aromatic rings or fused or otherwise conjugated derivatives thereof.

Preferably, at least one of  $\text{Ar}^1$ ,  $\text{Ar}^2$  or  $\text{Ar}^3$  is substituted by alkyl, alkoxy, ether, halo alkyl, amino alkyl, aryl or heteroaryl, where any alkyl group is a straight or branched chain of 1-10 carbon atoms. In a particularly preferred embodiment, at least one of  $\text{Ar}^1$ ,  $\text{Ar}^2$  or  $\text{Ar}^3$  is twice substituted with a straight or branched alkyl chain of 1-10 carbon atoms, for example octyl.

Preferably, at least one of  $\text{Ar}^1$ ,  $\text{Ar}^2$  or  $\text{Ar}^3$  is substituted in the ortho- or para-position by an alkoxy group, most preferably in the para- position. Suitably, the alkoxy group is a short chain alkoxy group, for example from 1 – 4 carbon atoms, and most preferably methoxy. Although not wishing to be bound by any theory, it is thought that the presence of a short chain alkoxy group in the para- position increases the ease of oxidation of the material, thus facilitating hole conduction.

Preferably, at least one of  $Ar^1$ ,  $Ar^2$  or  $Ar^3$  is selected from structures (i) to (xii)



wherein  $R^1$  and  $R^2$  are independently selected from, hydrogen, halogen, alkyl, alkoxy, ether, amino alkyl, aryl or heteroaryl, in which any alkyl group is straight or branched chain of 1 to 10 carbon atoms; wherein  $n$  is an integer; and wherein any of (i) to (xii) may be substituted or unsubstituted.

These materials exhibit high conductivities due to the presence of an extended conjugated structure. Preferably, the material exhibits extended  $\pi$  or mixed  $\pi$ -lone pair

conjugation. This may be for example, by way of Ar-N-Ar type linkages, where the Ar groupings may themselves comprise extended conjugation through the connection of aromatic ring moieties with unsaturated groups.

Alternatively, [Y] may comprises other organic materials which provide both electronic charge transport properties and can be derivatised to include ion-chelating groups. Some non-limiting examples include poly(1,4-phenylene), polypyrrole, poly(p-phenylenevinylene) (PPV), poly(thiophene), MEH-PPV, polyaniline and PEDOT.

Preferably, X comprises at least one group selected from:  $[-(\text{CH}_2\text{CH}_2\text{O})_n\text{CH}_2\text{CH}_2\text{OCH}_3]$ ,  $[-(\text{OCH}_2\text{CH}_2)_n\text{OCH}_3]$ ,  $[-(\text{CH}_2\text{CH}(\text{R})\text{O})_n\text{CH}_2\text{CH}_2\text{OCH}_3]$  and  $[-(\text{OCH}(\text{R})\text{CH}_2)_n\text{OCH}_3]$ ; wherein n is an integer, preferably 2 to 10, more preferably 2 to 4; wherein R is straight or branched alkyl chain of 1 to 10 carbon atoms, preferably of 1 or 2 carbon atoms.

The above ion-chelating groups are based on the repeat unit  $[-\text{OCH}_2\text{CH}_2-]$ . Side chain branching and/or the inclusion of  $[-\text{OCH}_2\text{O}-]$  repeat units, are advantageous to inhibit crystallisation after metal ion complexation. The groups contain preferably 3 or more  $[-\text{OCH}_2\text{CH}_2-]$  units and most preferably 3 units terminating in OR' (R' = alkyl of up to 10 carbon atoms, e.g. methyl) containing 4 oxygen atoms for ion-chelation. Other ion-chelating groups may be made according to the specific need for ion binding, some examples including crown ethers, podands, lariat ethers, cryptands and spherands.

Although not as effective, a group with the structure of an ion-chelating group may be used as a linking group between moieties of general formula (II). If such a group is used it should be in the ortho- or para- position and not in the meta- position. Most preferably if such a linking group is used, it is in the para- position.

Suitably, the at least one ionic species is chosen from:  $\text{Li}^+$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Cs}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$  or any other suitable ions. These may be provided for example, from triflimide, halides, perchlorates, trilates and BARF salts of the above cations.



In an embodiment of the present invention, the conducting material comprises an ohmic conductor. Suitable are metals such as gold, aluminium, copper, platinum, silver and calcium, non-metals such as graphite, highly-doped semiconductors such as ITO, fluorine-doped tin oxide, aluminium-doped zinc oxide and organic conductors such as PEDOT-PSS and polyaniline.

In an alternative embodiment, the conducting material comprises a semiconducting material. Suitable are  $\text{TiO}_2$ ,  $\text{ZnO}$ ,  $\text{SnO}$ ,  $\text{Ta}_2\text{O}_5$ ,  $\text{Nb}_2\text{O}_5$ ,  $\text{WO}_3$ , OMeTAD, PPV, Cu-phthalocyanin, oligo- or polythiophenes, polypyrroles, TPDs, pentacene and perylenes.

In a further alternative embodiment, the conducting material comprises an ionic conductor. Suitable are polymer electrolytes such as PEO, co-polymers comprising PEO for example, poly-epichlorohydrin-co-ethyleneoxide and polymers supporting redox active species such as  $\text{Ru(II)/(III)}$  and  $\text{Co(II)/(III)}$ .  $\text{C}_{60}$  and its derivatives may also be suitable.

The semiconductor may be an inorganic semiconductor such as  $\text{TiO}_2$ ,  $\text{ZnO}$ ,  $\text{SnO}$ ,  $\text{Ta}_2\text{O}_5$ ,  $\text{Nb}_2\text{O}_5$ ,  $\text{WO}_3$  or an organic semiconductor such as OMeTAD, PPV, Cu-phthalocyanin, oligo- or polythiophenes, polypyrroles, TPDs, pentacene and perylenes. In a preferred embodiment, the semiconductor is a nano-crystalline metal oxide for example, a nano-crystalline titania film which may be sensitised. Suitable sensitisers include dyes based on ruthenium bipyridyl complexes or organic dyes such as coumarins.

The semiconductor may be porous, in which case preferably, the dual-function material is at least partially contained within the pores of the semiconductor. This maximises the surface area of the semiconductor that is in contact with the dual-function material.

The composite structures of the present invention are particularly suitable for inclusion in electrochemical devices and thus in accordance with a second aspect of the

further, or two ohmic conductors such that the device is provided with two external ohmic conductors. Dependent on the design of a particular device, the ohmic conductors may be arranged such that they are in direct contact with the outer surfaces of the composite structure or there may be one or more additional intervening layers.

5

Preferably, the ohmic conductors comprise metallic conductors such as gold, aluminium, copper, platinum, silver and calcium, or non-metallic conductors such as graphite, highly-doped semiconductors such as ITO, fluorine-doped tin oxide, aluminium-doped zinc oxide or organic conductors such as PEDOT-PSS and polyaniline

10 Both ohmic conductors may be the same or different.

15

The composite structure of the present invention may be incorporated into a photo-voltaic cell however, its use is not limited thereto. Other potential applications will be known to those skilled in the art and include photodiodes, batteries, electrodes, electrochromic devices and light-emitting diodes.

The invention will now be described by way of example only and with reference to the following drawings in which:

20

Figure 1 is a schematic diagram of an example of a composite structure according to the present invention;

Figure 2 is a schematic diagram of a further example of a composite structure according to the present invention;

25

Figure 3 is a schematic diagram of a photo-voltaic cell incorporating a composite structure according to the present invention; and,

30

Figure 4 is a graph comparing the current-voltage characteristics of the photo-voltaic cell of Fig. 3 with a similar device incorporating a structure absent a dual-function material.

With reference to Fig. 1, a composite structure comprises a dual-function material 1 intermediate an electron-transporting semiconductor 2 and a hole-conducting semiconductor 3. In the alternative embodiment of Fig. 2, the electron-transporting semiconductor is replaced with a metal layer 4.

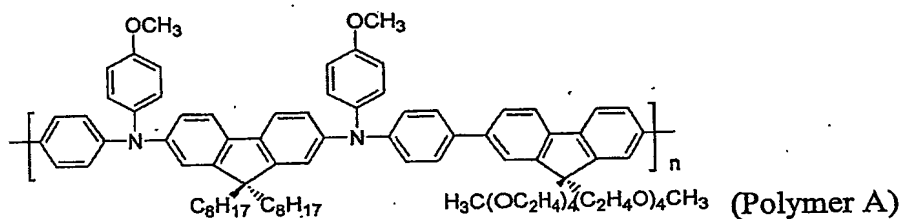
5

### EXAMPLE 1

#### Fabrication of a dye sensitised photo-voltaic cell.

With reference to Fig. 3, a dye sensitised nanocrystalline  $\text{TiO}_2$  film 3 was prepared on a glass substrate 6 using the following procedure. The glass substrate was provided with a conducting coating of fluorine-doped tin oxide 5. A  $\text{TiO}_2$  paste, consisting of ca. 15nm sized particles (as determined by HRTEM) was prepared from a sol-gel colloidal suspension containing  $\text{TiO}_2$  particles (12.5 wt%) and Carbowax<sup>TM</sup> 20,000 (6.2wt%). The titania particles were produced by injecting titanium iso-propoxide (20ml) into glacial acetic acid (5.5g) under an atmosphere of argon followed by stirring for 10 minutes. The mixture was then injected into 0.1M nitric acid (120 ml) under an anhydrous atmosphere at room temperature and stirred vigorously. The resultant solution was left uncovered and heated at 80°C for 8 hours. After cooling, the solution was filtered using a 0.45  $\mu\text{m}$  syringe filter, diluted to 5wt%  $\text{TiO}_2$  by the addition of water and then heated in an autoclave at 220°C for 12 hours. The resultant colloid was re-dispersed with a 60 second cycle burst from a LDU Soniprobe horn and then concentrated to 12.5% by rotary evaporation. Carbowax<sup>TM</sup> 20,000 was added and the resulting paste was stirred slowly overnight to ensure homogeneity. The paste was spread onto the glass substrate with a glass rod, using adhesive tape as a spacer. The film was dried in air and then sintered at 450°C for 20 minutes, also in air. The thickness of the  $\text{TiO}_2$  film was ca. 3 $\mu\text{m}$ . The  $\text{TiO}_2$  film was sensitized by immersing it in a 1mM solution of a  $\text{RuL}_2(\text{NCS})_2$  dye in 1:1 acetonitrile/tert-butanol. Rinsing in ethanol removed any unadsorbed dye. Prior to use, samples were stored in dry glove box in the dark

30 A layer of a dual function material 1 was then deposited as follows. A solution was prepared by dissolving polymer A (structure below) and lithium triflimide, at a mole ratio of 1:12, in a chlorobenzene / acetonitrile solvent mixture (1:9 volume ratio).

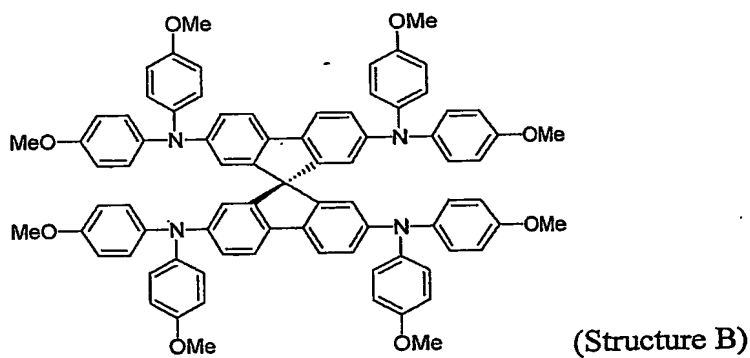


5 The dye sensitised TiO<sub>2</sub> film as prepared above was immersed in the polymer A solution for 2 hours at a temperature of 70 °C. The immersion time and temperature provide a control of the ion / polymer concentration at the interface. This step resulted in the conformal deposition of a layer of the dual function material on the surface of the dye sensitised, nanocrystalline TiO<sub>2</sub> film.

10

A hole-transporting semiconducting layer 2 of a spiro-OMeTAD material (structure B below) was then deposited onto the layer of dual-function material by spin coating from solvent solution (0.2M solution in chlorobenzene for 60 seconds). This solution did not contain any added ionic species, chemical dopants or ion-solvating species. The resulting sample was left under vacuum for 2 hours and then transferred to a thermal evaporator. A gold contact 7 was deposited under a pressure of ca.  $1-2 \times 10^{-6}$  atm. This provided a photo-voltaic cell 8 incorporating an composite structure according to the invention. For comparative purposes, a second device (not shown) was prepared omitting the layer of dual-function material.

20



**EXAMPLE 2****Cell Testing**

Both devices prepared in Example 1 had a cell area of  $0.2\text{cm}^2$  and were exposed to  $10\text{ mWcm}^{-2}$  of simulated AM1.5 solar irradiation during data collection, as indicated by arrow 9. As shown in Fig. 4, the current - voltage characteristics of the device according to the invention (curve 10) showed an efficient photovoltaic response. By contrast, the device absent the layer of dual-function material (curve 11) showed a negligible photovoltaic response.

The specific ordering of the layers in the device was found to be important. Surprisingly, reversing the order of the dual-function material (polymer A) and the hole-transporting semiconducting layer (structure B), produced a device which showed only a very poor photovoltaic response. This observation indicates that the dual-function material should be inserted at the interface of the dye-sensitised titania layer with the hole-transporting semiconducting layer. Although not wishing to be bound by any theory, it is thought to be advantageous that the dual-function material be present as a thin layer at the interface.

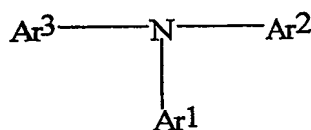
CLAIMS

1. A composite structure, the structure comprising a dual-function material intermediate a conducting material and a semiconductor; wherein the dual-function material comprises an organic material and at least one ionic species; wherein the organic material has both electronic charge transport properties and supports or chelates the at least one ionic species; and wherein the conducting material comprises an ohmic conductor, a semiconducting material or an ionic conductor.
2. A structure according to claim 1, wherein the organic material comprises at least one moiety represented by the general formula (I):



- wherein [Y] comprises an organic semiconductor; and wherein X comprises an ion-chelating group.

3. A structure according to claim 2, wherein [Y] comprises a moiety represented by the general formula (II):



(II)

- wherein Ar<sup>1</sup>, Ar<sup>2</sup> and Ar<sup>3</sup> are independently substituted or unsubstituted aromatic or hetero-aromatic rings or fused or otherwise conjugated derivatives thereof.

4. A structure according to claim 2, wherein [Y] comprises poly(1,4-phenylene), polypyrrole, poly(p-phenylenevinylene) (PPV), poly(thiophene), MEH-PPV, polyaniline or PEDOT

5. A structure according to any of claims 2 to 4, wherein X comprises at least one group selected from:  $[-(\text{CH}_2\text{CH}_2\text{O})_n\text{CH}_2\text{CH}_2\text{OCH}_3]$ ,  $[-(\text{OCH}_2\text{CH}_2)_n\text{OCH}_3]$ ,  $[-\text{CH}_2\text{CH}(\text{R})\text{O})_n\text{CH}_2\text{CH}_2\text{OCH}_3]$  and  $[-(\text{OCH}(\text{R})\text{CH}_2)_n\text{OCH}_3]$ ; wherein n is an integer, preferably 2 to 10, more preferably 2 to 4; wherein R is straight or branched alkyl chain of 1 to 10 carbon atoms, preferably of 1 or 2 carbon atoms.
6. A structure according to any of claims 2 to 4, wherein X comprises a crown ether, a podand, a lariat ether, a cryptand or a spherand.
7. A structure according to any preceding claim, wherein the at least one ionic species is chosen from:  $\text{Li}^+$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Cs}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ , triflimide, halide, perchlorate, trilate and BARF salts of the above cations.
8. A structure according to any preceding claim, wherein the conducting material comprises an ohmic conductor and is chosen from: a metal, graphite, a highly-doped semiconductor and an organic conductor.
9. A structure according to any of claims 1 to 7, wherein the conducting material comprises a semiconducting material and is chosen from:  $\text{TiO}_2$ ,  $\text{ZnO}$ ,  $\text{SnO}$ ,  $\text{Ta}_2\text{O}_5$ ,  $\text{Nb}_2\text{O}_5$ ,  $\text{WO}_3$ , OMeTAD, PPV, Cu-phthalocyanin, polythiophenes, polypyrroles, pentacene and perylenes..
10. A structure according to any of claims 1 to 7, wherein the conducting material comprises an ionic conductor and is chosen from: a polymer electrolyte, a polymer supporting a redox active species.
11. A structure according to any preceding claim, wherein the semiconductor is chosen from  $\text{TiO}_2$ ,  $\text{ZnO}$ ,  $\text{SnO}$ ,  $\text{Ta}_2\text{O}_5$ ,  $\text{Nb}_2\text{O}_5$ ,  $\text{WO}_3$ , OMeTAD, PPV, Cu-phthalocyanin, oligo- or polythiophenes, polypyrroles, TPDs, pentacene and perylenes.
12. A structure according to any preceding claim, wherein the semiconductor is porous and the dual-function material is at least partially contained within the pores of the semiconductor.

12. A structure according to any preceding claim, wherein the semiconductor is porous and the dual-function material is at least partially contained within the pores of the semiconductor.

5 13. An electrochemical device, the device comprising a structure according to any preceding claim and one further, or two ohmic conductors such that the device is provided with two external ohmic conductors.

10 14. A photo-voltaic cell, the cell comprising a structure according to any of claims 1 to 12.

15 15. A device according to claim 13 which is a photodiode, a battery, an electrode, an electrochromic device or a light-emitting diode.



**COMPOSITE STRUCTURE****Abstract**

A composite structure comprises a dual-function material intermediate a  
10 conducting material and a semiconductor. The dual-function material comprises an  
organic material and at least one ionic species such that the organic material has both  
electronic charge transport properties and supports or chelates the at least one ionic  
species. The conducting material comprises an ohmic conductor, a semiconducting  
15 material or an ionic conductor. The composite structures are suitable for use in  
electro-chemical devices such as photo-voltaic cells, photodiodes, batteries, electrodes,  
electrochromic devices and light-emitting diodes.

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Patented May 11, 1966

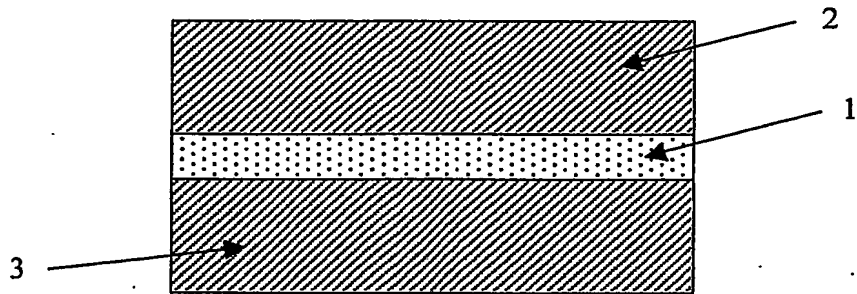


Fig. 1

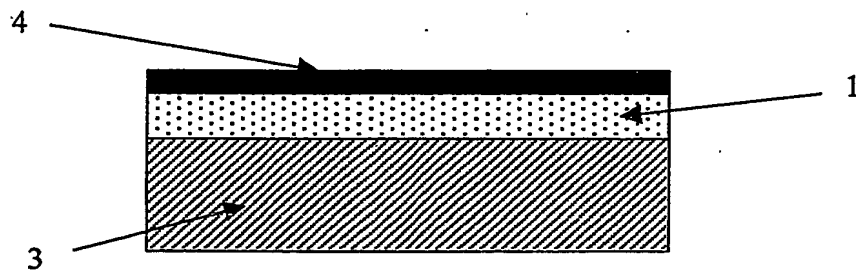


Fig. 2

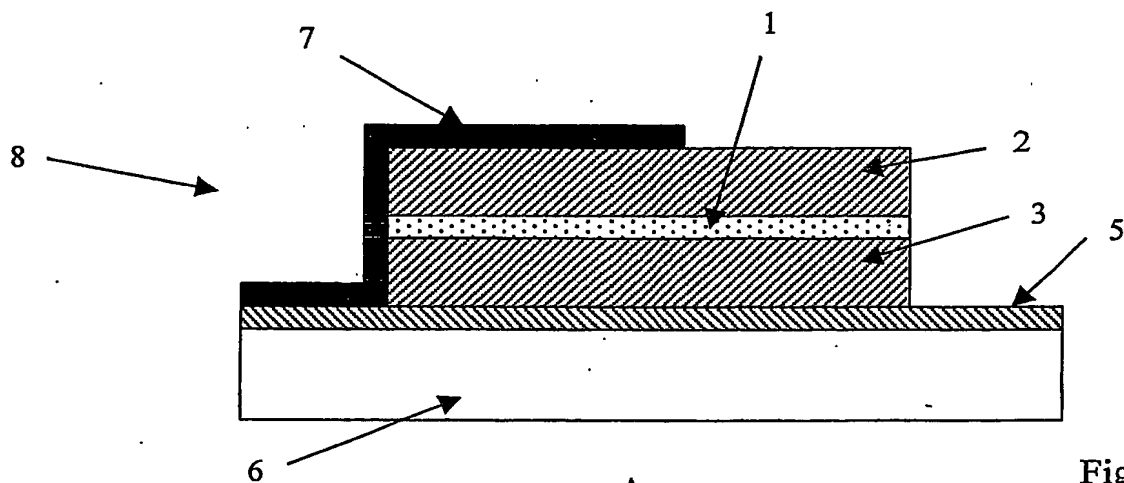


Fig. 3

1. The first step is to identify the problem or question that needs to be answered. This involves understanding the context and the specific requirements of the task.



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